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Characterization of a Polymer Composite Section of Foreign Armor

by James M. Sloan, Seth R. Ghiorse,
Donovan Harris, and Gumersindo Rodriguez

ARL-TR-2158 March 2000

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ARL-TR-2158 **March 2000**

Characterization of a Polymer Composite Section of Foreign Armor

James M. Sloan, Seth R. Ghiorse, Donovan Harris,
and Gumersindo Rodriguez
Weapons and Materials Research Directorate, ARL

Abstract

The chemical and physical characterization of a foreign polymer composite of Russian armor was performed. This report has identified and quantified the composition of an unknown cored section of Russian composite armor.

The material was identified as a woven glass in an epoxy matrix. The environmental scanning electron microscopy (ESEM) results determined that the reinforcing fiber is E glass, as opposed to S glass or the Russian equivalent of S glass, Vertex. E glass is inferior to S glass in both armor and structural applications; however, it is often chosen as a reasonable tradeoff because it is much more affordable. Fourier transform infrared (FT-IR) spectroscopy results identified the matrix to be an epoxy resin—likely a diglycidyl ether of bisphenol-A. Thermal gravimetric analysis (TGA) clearly showed that all specimens contain approximately 90% fiber and 10% organic resin by weight. The cored composite density was also examined. All density measurements were close to 2.0 g/cm^3, which is consistent with the typical density of a highly filled glass/polymer composite. It was found that the bulk and local section density values were approximately equal within a reasonable margin common in the composite materials industry. No designed-in density gradient vs. length was observed.

Acknowledgments

The authors wish to thank Mr. Jim Kleinmeyer for photographing the core specimens in their as-received state from the National Ground Intelligence Center (NGIC), which funded this work.

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1. Introduction

The National Ground Intelligence Center (NGIC) supplied a cylindrical, cored section of highly filled fibrous polymer composite Russian armor of unknown makeup to the U.S. Army Research Laboratory's Weapons Materials Division for analysis. At first look, the composite construction appeared to the eye to be a laminated orthotropic woven cloth. Its yellow color made it easy to mistake for an aramid composite, a material often used in armor applications; however, analysis showed that this was not the case. The composite is nondomestic E glass in an epoxy matrix.

2. Experimental

2.1 General. The cored specimen was delivered inside a sealed, tightly fitting plastic tube. For recordkeeping purposes, an initial photograph was taken of the core sample while still in the sample tube. The end caps of the sample container were then removed, and the rubber eraser end of a pencil was used to gently push the core sample through the tube and onto a piece of white paper. The size and packing order of the individual layers was then documented.

The as-received specimen arrived sliced into 21 distinct cross sections, ranging from wafer thin to two inches in length. Each piece was arranged on the paper in order, assigned a number, and photographed. Next, each was weighed, again for tracking purposes. The weight values were used as a means to uniquely identify each piece. The pieces had ragged edges, and small losses of fiber and matrix dust were noted.

For clarity in this document, layer 1 is arbitrarily identified as the first separable disc located at the "interior" of the NGIC core sample. The remaining layers were numbered in sequence from the interior (layer 1) to the exterior (layer 21) side.

2.2 Environmental Scanning Electron Microscopy (ESEM). ESEM was utilized to determine the composition of the reinforcing fiber in the composite sample. Three samples, designated interior (layer 2), middle (layer 7), and exterior (layer 16), were evaluated using the ESEM. The specimens were prepared by pyrolyzing the organic base resin at 400° C. The resultant residue consisted of only the inorganic reinforcing fiber. A sample of each specimen was mounted on an aluminum stub using conductive carbon tape and silver adhesive as an electron drain. The samples were imaged by the ESEM at 250× magnification, with an acceleration voltage of 20 keV, by means of lanthanum hexaboride crystal source. An elemental analysis was performed on each of the three layers using an EDAX light element energy dispersive spectrometer. Samples of domestic Owens-Corning E and S-2 glass fibers were first analyzed for reference purposes, along with a sample of Russian-made S glass sold under the brand name "Vertex." The appearance of oxygen in the ESEM data are considered not relevant because the imaging gas utilized in the sampling chamber during the ESEM analysis was water vapor. The oxygen atom present in the water vapor molecule masks the presence of oxygen in the resulting ESEM spectral data of the sample fibers. Both E- and S-type glasses have oxygen as a major compositional element. The samples were run at 3.5 torr.

2.3 Composite Density. Helium pycnometry was used to measure density. The instrument was a Quantachrome Ultrapycnometer 1000. This is a noninvasive procedure that uses purified helium as the displaced medium. In this method, the specimens are subjected to an initial pressure of about 18 psig in a sealed cell. After a precision reading of this pressure, a valve opens, allowing the helium to fill the connected expansion cell. This second pressure is then recorded. Through previous calibration of the instrument, these two pressure values are used in the working equation to calculate the volume of the specimen. The specimen weight, previously input into the pycnometer, is then used together with the volume measurement to calculate density. The 21 cored armor specimen cross sections were combined to form three subgroups of approximately equal volume.

After calibration, the specimens were placed in the measurement cell and purged with helium for 6 min. The automated measurement procedure then began, and 15 samplings were taken for

each data point. The mean value of these samplings is reported as the density. The following measurements were made:

(1) Bulk measurement of the entire cored specimen.

(2) Measurement of layers 1–11.

(3) Measurement of layers 12–13 (longer pieces).

(4) Measurement of layers 14–21.

(5) Measurement of darker-colored portions of layers 12 and 13 only (layer 13 had to be split into two pieces for this).

2.4 Thermal Gravimetric Analysis (TGA). TGA was used to determine the resin and fiber content. The instrument was a model 2950 Hi-Res TGA made by TA Instruments equipped with a 9900 thermal analyzer/computer for quick data reduction. TGA utilizes differences in weight as a function of temperature as materials become pyrolyzed to determine the individual components in the cured composite. A sample size of 10–20 mg was weighed and placed in an aluminum sample pan. The sample was scanned in the temperature range between 30° C and 980° C with a scanning rate of 10° C/min. A nitrogen purge was used as the carrier gas to minimize the production of oxidative products, yielding only molecular fragments of the organic resin and its components.

2.5 Fourier Transform Infrared (FT-IR) Spectroscopy. A Perkin Elmer FT-IR model Spectrum 2000 was used to obtain IR spectra. Samples of 25 mg were pyrolyzed in a specially designed cell at 450° C. The sampling unit allowed the pyrolyzate to be deposited onto a KBr salt, which was placed directly into the FT-IR spectrometer. The spectra were then recorded. This method was preferred over several others that were attempted because it eliminated the IR

spectrum of the reinforcing fibers. A total of 32 scans were co-added at a resolution of 4 cm^{-1}. Infrared spectra were recorded at various sections along the core specimen.

3. Results and Discussion

3.1 General. Optical photographs were used to determine and document the size of the entire core specimens and subsequent size and shape of the individual layers.

Figure 1 shows the photograph of the as-received core sample. The overall length was approximately 5 inches. The ragged edges resulting from the cutting process can clearly be seen along the length sample. Frayed ends from the reinforcing fiber are evident.

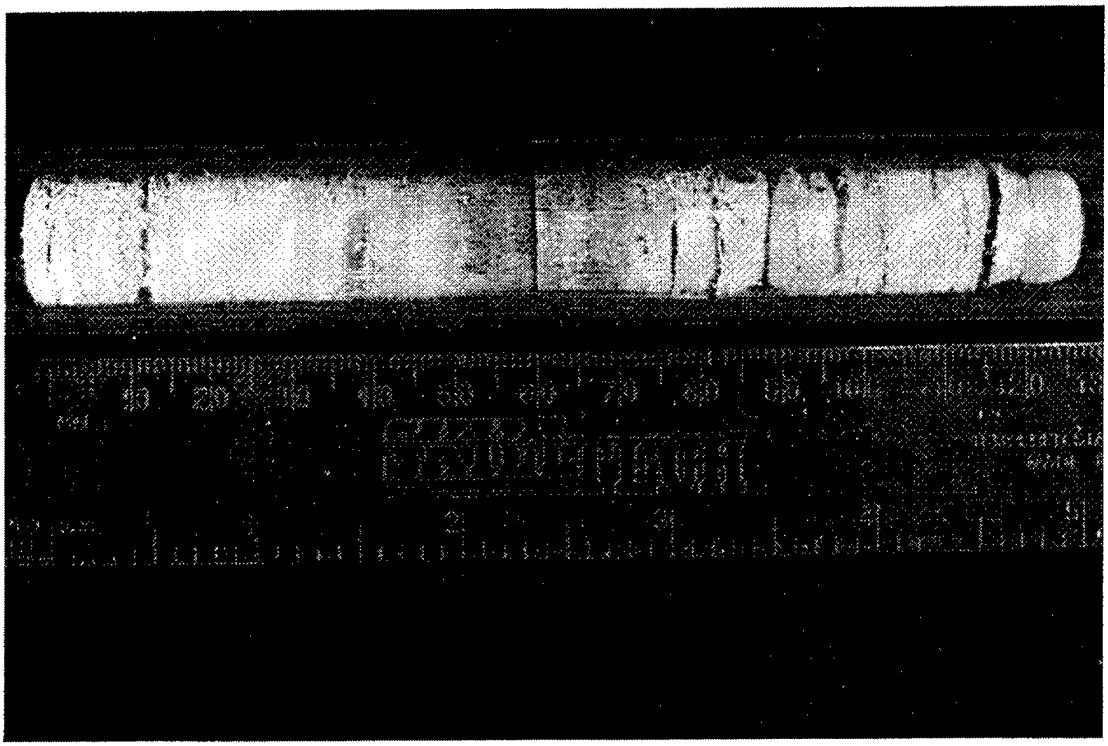


Figure 1. Photograph of the As-Received NGIC Russian Armor Core Sample.

Figure 2 shows the 21 individual core layers after removal from the protective plastic liner. The samples are circular and are approximately 5/8 inch in diameter. They were easily separated

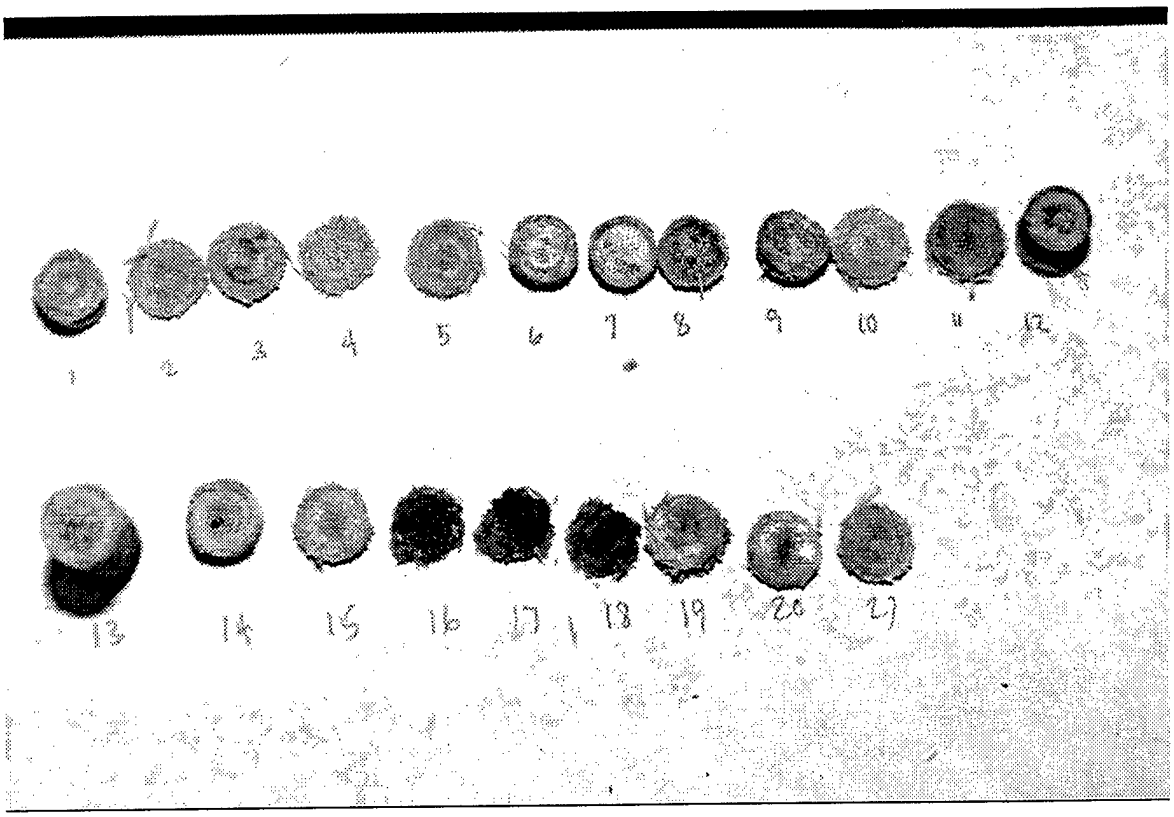
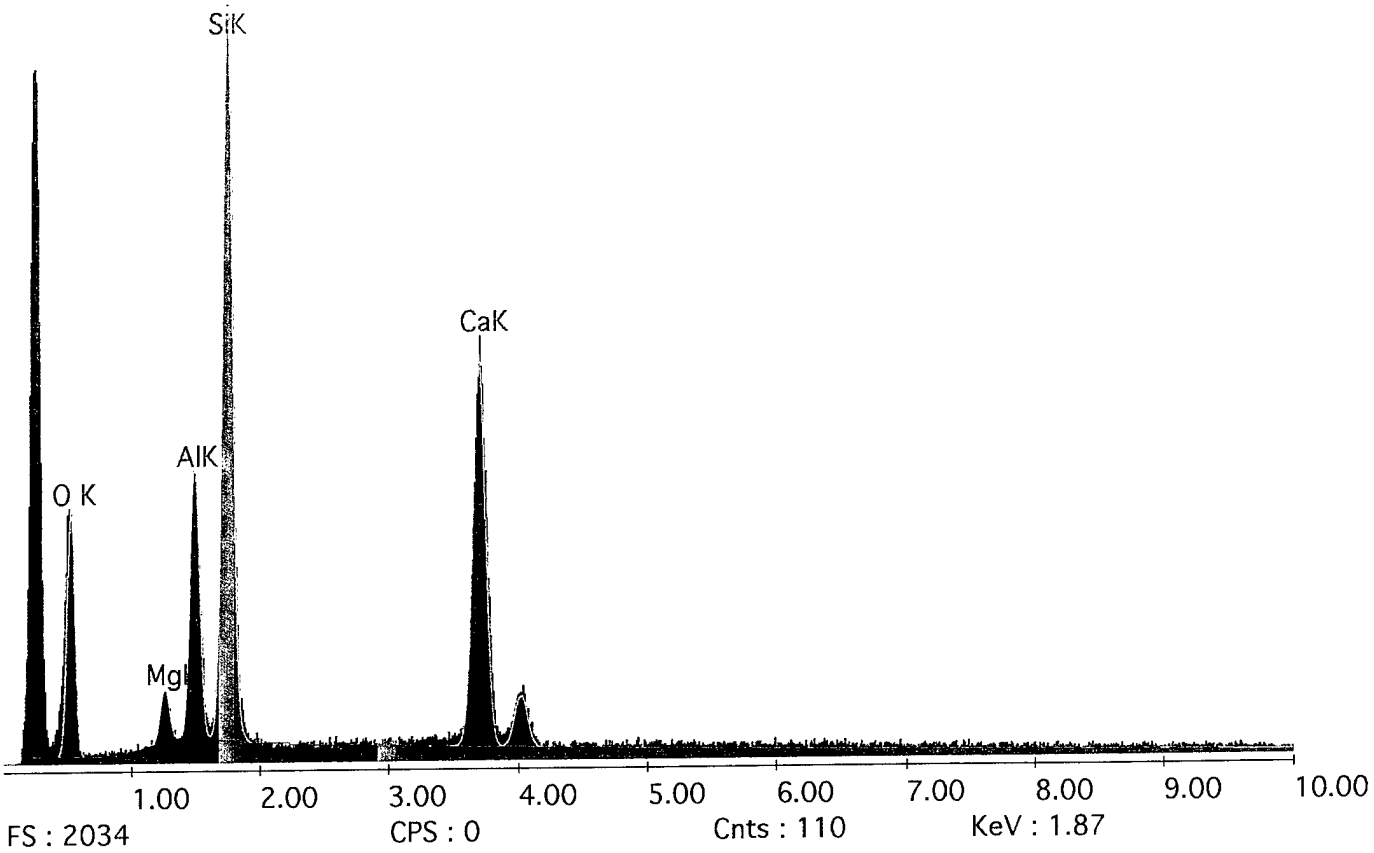


Figure 2. Photograph of the Individual NGIC Russian Armor Core Sample Layers.

from one another. The numbers correspond to their respective position away from the interior. Layer 1 corresponds to the 0-inch mark in Figure 1.

3.2 ESEM. ESEM was used to evaluate three individual layers along the core sample. These experiments were run to determine the reinforcing fiber composition. Figures 3, 4, and 5 show the resultant data for the three samples taken from the NGIC core sample. These correspond to layers 2, 7 and 16, respectively. Below each graph is an elemental quantitative analysis of the corresponding specimens. Examination of the specimen elemental analysis spectra showed little difference between the three layers.

To determine the specific type of reinforcing fiber, two well-known domestic glass fibers and one Russian glass fiber were examined for comparison to the unknown Russian armor fiber. The two domestic reference fibers were Owens-Corning E and S-2 glass. Their spectra are shown in



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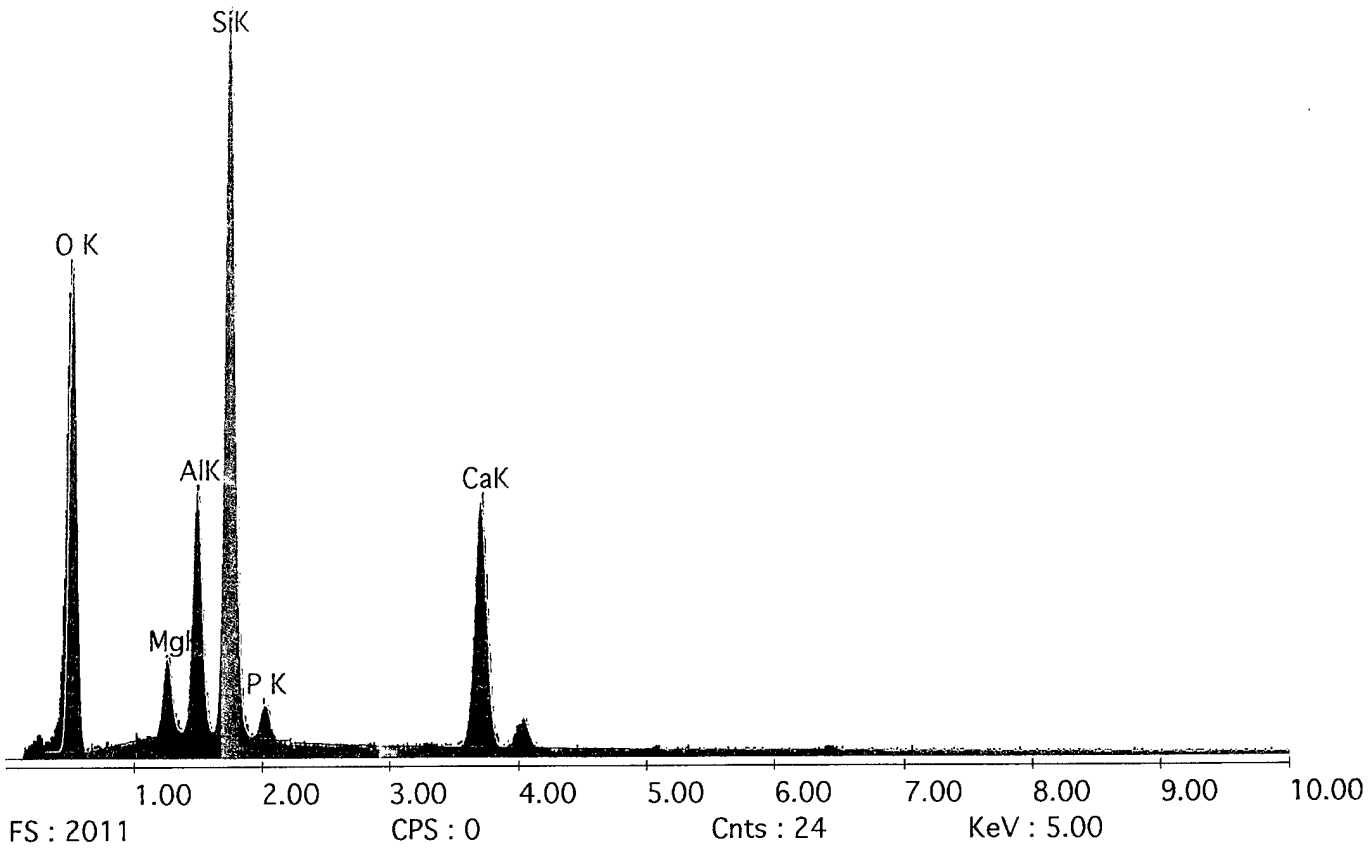
Element	K Ratio	Weight %	Atomic %
O K	0.2283	49.006	64.973
MgK	0.0255	2.551	2.226
AlK	0.1131	9.359	7.357
SiK	0.2900	21.058	15.904
CaK	0.3432	18.026	9.540
Total		100.000	100.000

Element	Net Inten	Backgrd	Inten Error	P/B
O K	51.10	1.72	1.60	29.68
MgK	12.20	6.38	3.97	1.91
AlK	67.56	8.23	1.45	8.21
SiK	179.34	6.90	0.86	26.00
CaK	123.99	5.46	1.03	22.73

Figure 3. ESEM From Layer 2 of the NGIC Russian Armor Core Sample.



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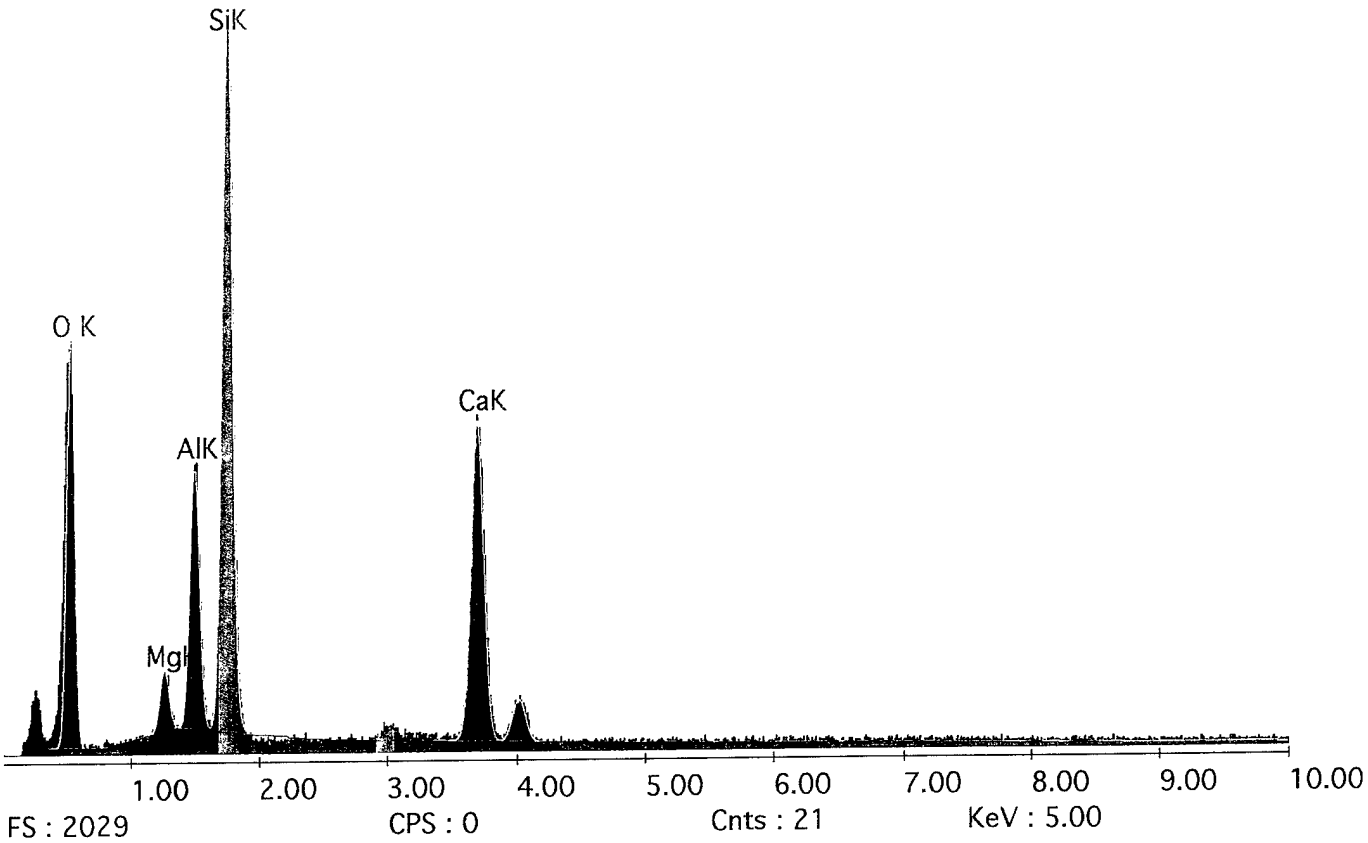
Element	K Ratio	Weight %	Atomic %
O K	0.4072	61.095	74.498
MgK	0.0330	3.211	2.576
AlK	0.0907	7.317	5.291
SiK	0.2597	17.616	12.236
P K	0.0149	1.125	0.708
CaK	0.1945	9.637	4.691
Total		100.000	100.000

Element	Net Inten	Backgrd	Inten Error	P/B
O K	117.43	2.09	1.12	56.27
MgK	20.36	8.39	3.17	2.43
AlK	69.75	9.96	1.54	7.01
SiK	206.94	7.94	0.85	26.06
P K	10.72	7.29	4.76	1.47
CaK	90.54	4.70	1.30	19.28

Figure 4. ESEM From Layer 7 of the NGIC Russian Armor Core Sample.



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Element	K Ratio	Weight %	Atomic %
O K	0.3504	58.376	72.662
MgK	0.0274	2.680	2.195
AlK	0.0997	8.022	5.921
SiK	0.2657	18.182	12.892
CaK	0.2568	12.740	6.330
Total		100.000	100.000

Element	Net Inten	Backgrd	Inten Error	P/B
O K	81.72	1.64	1.23	49.88
MgK	13.66	6.60	3.62	2.07
AlK	62.05	8.13	1.48	7.63
SiK	171.18	6.49	0.85	26.36
CaK	96.65	3.92	1.14	24.68

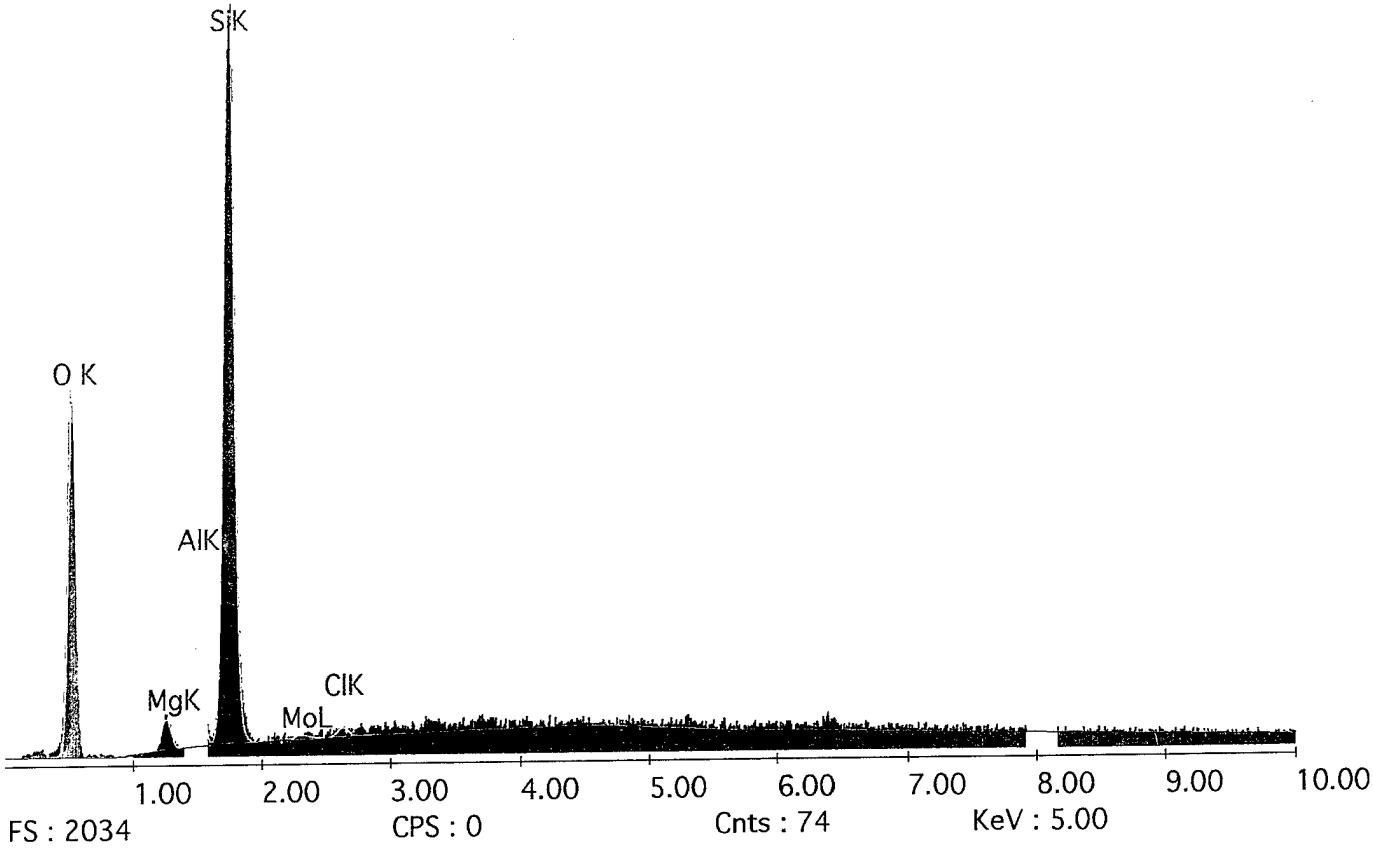
Figure 5. ESEM From Layer 16 of the NGIC Russian Armor Core Sample.

Figures 6 and 7, respectively. The Russian S glass spectra is shown in Figure 8. These known ESEM spectra were compared to that of the unknown core specimen. Close examination of Figures 3, 4, and 5 show the presence of three major elements—silicon (Si), aluminum (Al), and calcium (Ca)—with a satellite band representing lesser amounts of magnesium (Mg). The spectra of the reference S-2 glass (Figure 6) show the presence of Si, Al, and a smaller amount of Mg. No Ca was detected. Figure 7 shows the reference spectrum of the domestic E glass. The spectrum appears to have all the primary spectral features of the unknown glass fibers. The presence of Si, Al, Ca, and Mg are evident. Table 1, taken from Lubin (1982),[1] documents the elemental composition of domestic E glass and S glass. Table 1 also documents the presence of boron (B) as a component in E glass. In our ESEM data, the B peak should appear just below the oxygen (O) peak. While a small peak does exist in all the ESEM spectra, these peaks are more likely due to the formation of ice crystals in the sample chamber. This is a direct result of the environmental air in the chamber. Hence, the confirmation of the presence of B is not possible from these data.

Of significant note in Table 1 is the absence of Ca in the S glass material; however, E glass contains a considerable amount (17%). A clear band attributable to Ca is evident in the ESEM data of the unknown fibers (Figures 3–5). This is direct evidence for the identification of E glass as the reinforcing fiber present in the composite.

3.3 Composite Density. Table 2 lists the results of the density testing. The composite edges were highly frayed, and small amounts of loose fiber and resin fell off. Nevertheless, a good portrayal of the density was obtained. Highly filled polymer composite density values near 2 g/cm^3 are typical of glass-reinforced composites. The density values ranged from a high value of 2.17 g/cm^3 to a minimum of 1.97 g/cm^3. This represents a span of approximately 9%. No trends were observed, and it is logical to conclude from these results that the cored specimen has no designed-in density variation along its length. The 0.2 g/cm^3 measured spread in density is minor and is not uncommon in composites. Several factors may cause this effect These

[1] Lubin, George (ed.). *Handbook of Composites*. New York: Van Nostrand Reinhold Company, p. 139, 1982.



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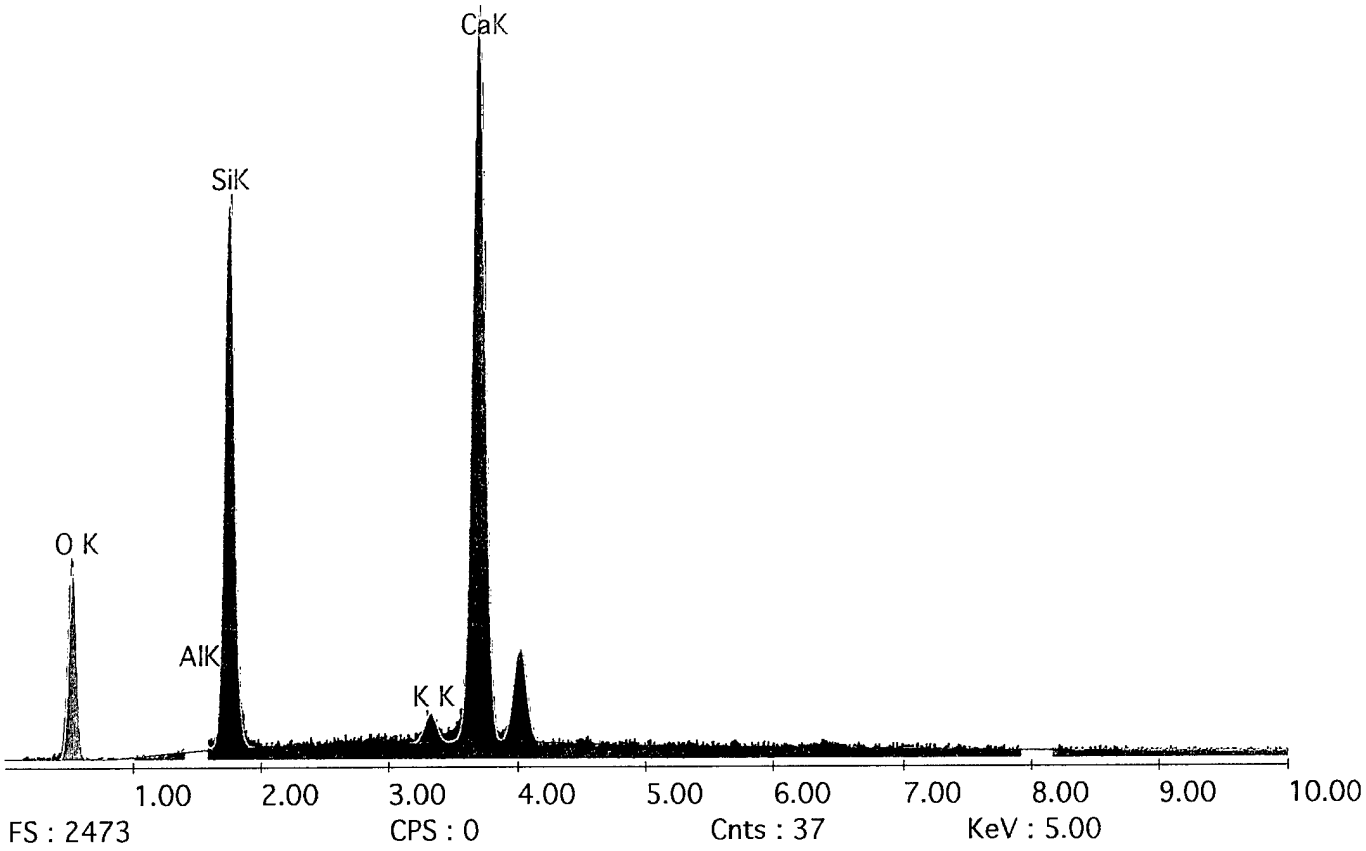
PEI User Set : 3, Elements

Element	K Ratio	Weight %	Atomic %
O K	0.4580	60.290	72.662
MgK	0.0183	1.796	1.425
AlK	0.1012	8.081	5.775
SiK	0.4076	28.780	19.759
MoL	0.0077	0.565	0.113
ClK	0.0071	0.488	0.266
Total		100.000	100.000

Element	Net Inten	Backgrd	Inten Error	P/B
O K	45.83	0.06	1.29	756.12
MgK	4.20	1.18	4.81	3.55
AlK	28.94	2.03	1.67	14.25
SiK	116.79	2.49	0.81	46.86
MoL	0.80	3.39	22.18	0.24
ClK	1.55	3.93	13.22	0.39

Figure 6. ESEM From Owens-Corning S-2 Glass Reference.



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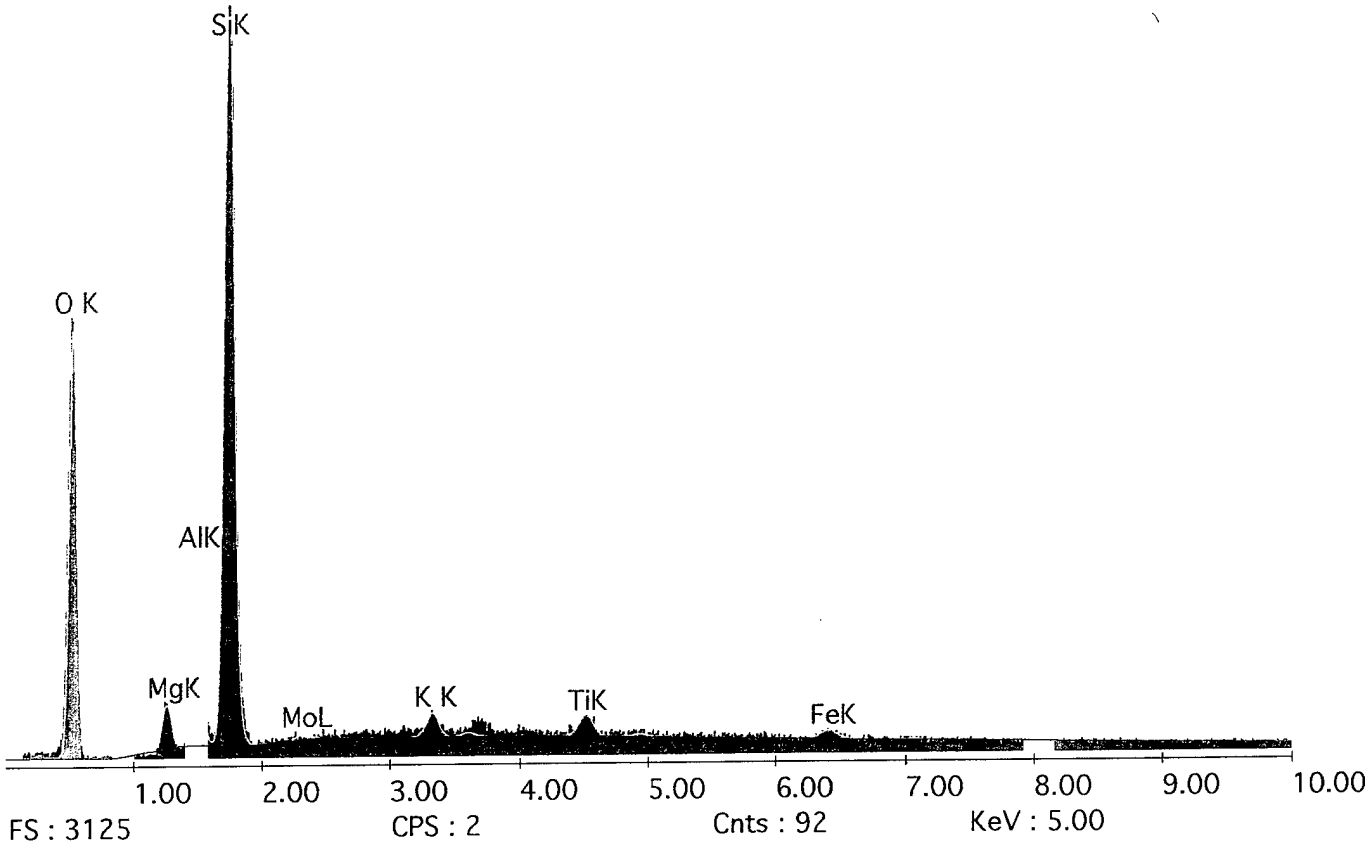
ZAF Quantification Method
PEI User Set : 3, Elements

Element	K Ratio	Weight %	Atomic %
O K	0.1668	48.937	67.483
AlK	0.0327	3.059	2.502
SiK	0.2101	15.213	11.950
K K	0.0209	1.135	0.640
CaK	0.5695	31.656	17.425
Total		100.000	100.000

Element	Net Inten	Backgrd	Inten Error	P/B
O K	85.82	0.00	1.61	85.82
AlK	48.09	5.96	2.28	8.07
SiK	309.42	7.64	0.86	40.48
K K	20.69	13.38	4.21	1.55
CaK	506.49	13.87	0.67	36.53

Figure 7. ESEM From Owens-Corning E Glass Reference.



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ZAF Quantification Method
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Element	K Ratio	Weight %	Atomic %
O K	0.4627	61.415	74.239
MgK	0.0238	2.382	1.895
AlK	0.0920	7.531	5.398
SiK	0.3456	24.380	16.788
MoL	0.0065	0.453	0.091
K K	0.0204	1.150	0.569
TiK	0.0279	1.546	0.624
FeK	0.0210	1.143	0.396
Total		100.000	100.000

Figure 8. ESEM From Known Russian S Glass Reference (Vertex Brand).

Table 1. Composition of Domestically Produced E Glass and S Glass

	E Glass (Weight-Percent)	S Glass (Weight-Percent)
Silicon Oxide	54.3	64.2
Aluminum Oxide	15.2	24.8
Ferrous Oxide	—	0.21
Calcium Oxide	17.2	0.01
Magnesium Oxide	4.7	10.27
Sodium Oxide	0.6	0.27
Boron Oxide	8.0	0.01
Barium Oxide	—	0.2

Source: Lubin, George (ed.). *Handbook of Composites.* New York: Van Nostrand Reinhold Company, p. 139, 1982.

Table 2. Density Results for NGIC Russian Armor Samples

Cross-Section I.D.	Approximate Length (inches)	Composite Density (g/cm^3)
Layers 1–21 (Bulk)	5.0	2.11
Layers 1–11	2.0	2.17
Layers 12–13	1.5	2.03
12–13, Dark Colored Portion Only	1.0	1.97
Layers 14–21	1.5	2.14

include the loss of fraying fibers and matrix resin, a coring process that seems to have been harsh on the specimen, causing bare fiber in some areas and possibly compressing the composite itself (which would make it more dense in some locations), as well as typical variations associated with composite manufacture.

3.4 TGA. Thermal methods of analysis measure the change in physical or chemical property as a function of temperature. Modern thermal analysis has proven extremely useful in the compositional analysis of complex polymer mixtures, such as composite materials and rubber compounds. TGA analysis utilizes the thermal degradation characteristics of polymer materials

to measure the weight loss as a function of temperature. The resultant data allow one to quantitatively determine the individual amounts of polymer, filler, and small organic additives.

Samples were taken from layers 2, 7, and 16, and TGA experiments were carried out. Figure 9 shows a TGA experiment for these three separate specimens taken along the NGIC core sample. These three samples were selected as representative specimens of the interior, middle, and exterior of the entire core sample. A major transition occurred at approximately 400° C. This corresponds to the degradation of the base resin. From these data, we can calculate the respective amounts of base resin and reinforcing fiber. Analysis of the data show that the middle layer (no. 7) is richer in resin content than either of the two outer layers. The resin content for layer 2 was calculated as 5.0%, while the resin content for layer 16 is 8.1%. Table 3 shows the respective compositions for the three specimens.

3.5 FT-IR Spectroscopy. Initial experiments on the entire separate samples yielded FT-IR spectra that exhibited very little absorbance bands attributable to the organic base resin. This was caused by the very high reinforcing inorganic fiber content as noted in Table 3. It was decided that pyrolyzing the specimen to separate the resin from fiber would yield interpretable spectra.

Figures 10–12 show the pyrolosis FT-IR spectra for three separate specimens taken along the NGIC core sample. These are identified as layers 2, 7, and 16, respectively, and are the same specimens evaluated in the microscopy and thermal analysis section of this report. All three spectra appear to be identical with respect to chemical structure.

Examination of the FT-IR spectra confirms the identity of the base resin as an epoxy. Significant IR bands at 1,500 cm^{-1} and 1,600 cm^{-1} are characteristic of the phenyl rings located along the backbone of the epoxy resin. IR spectral bands at 3300 cm^{-1} and in the 1,000–1,200-cm^{-1} region are indicative of the curing process, as these can be assigned to the C-O-H and C-O stretches in the cured resin. The 1,725 cm^{-1} band is characteristic of the C=O stretch, another resultant structure due to the curing process. While absolute identification is

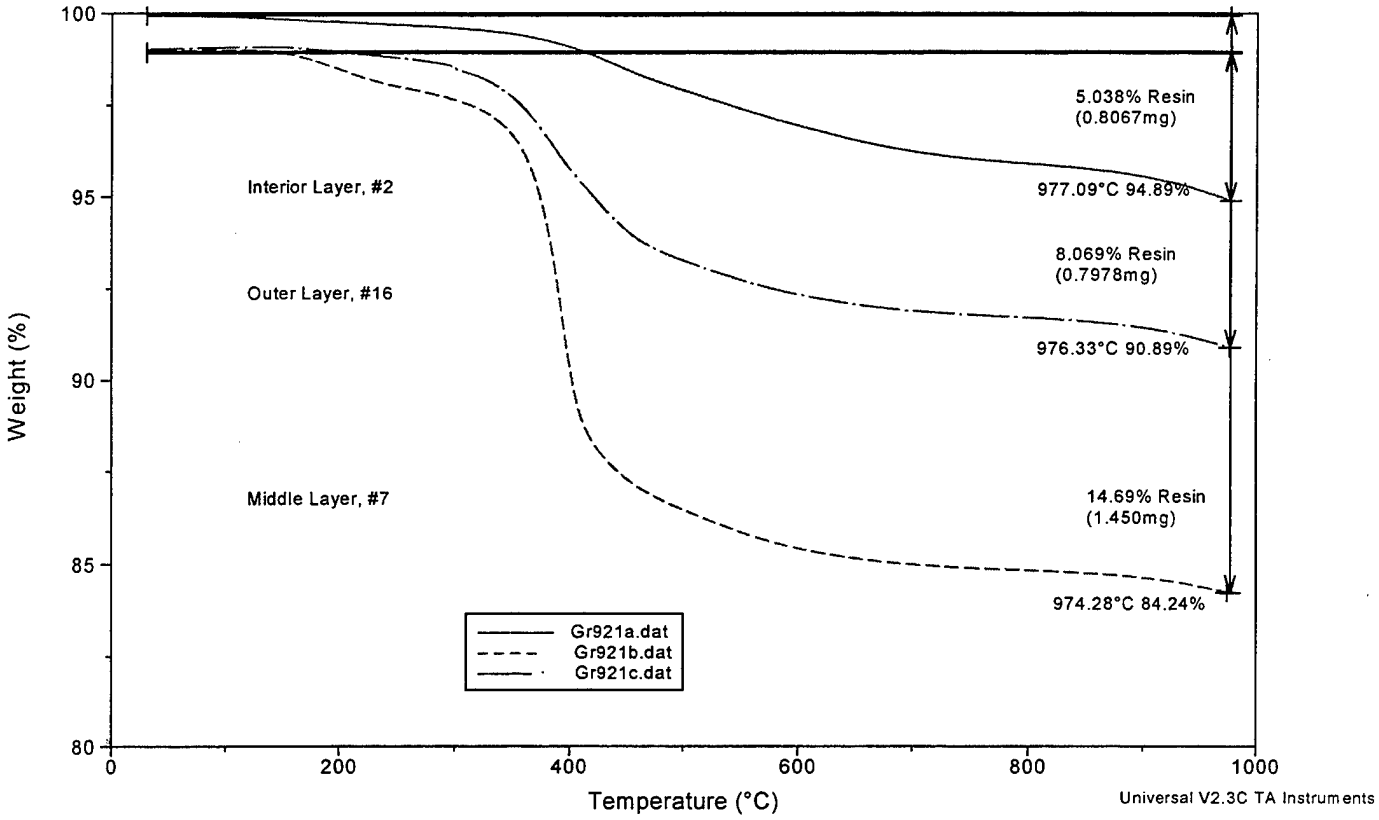

Figure 9. TGA Experiment Showing Degradation of Layers 2, 7, and 16.

Table 3. Quantitative Results Derived From TGA Data

Layer No.	Resin Content (Weight-Percent)	Fiber Content (Weight-Percent)
2	5.0	95.0
7	14.7	85.3
16	8.1	91.9

difficult, the resin appears to be a diglycidyl ether of bisphenol-A. This is a very common and inexpensive class of epoxy resin and is widely available on the world market.

The spectral features in the 1,550-cm^{-1} and 1,380-cm^{-1} bands are likely due to a diamine-type curing agent. The exact curing agent is even more difficult to determine as there are a large number of possibilities. However, based on the FT-IR spectra and the type of resin, it is feasible to suggest that a diamine-type curing agent was used.

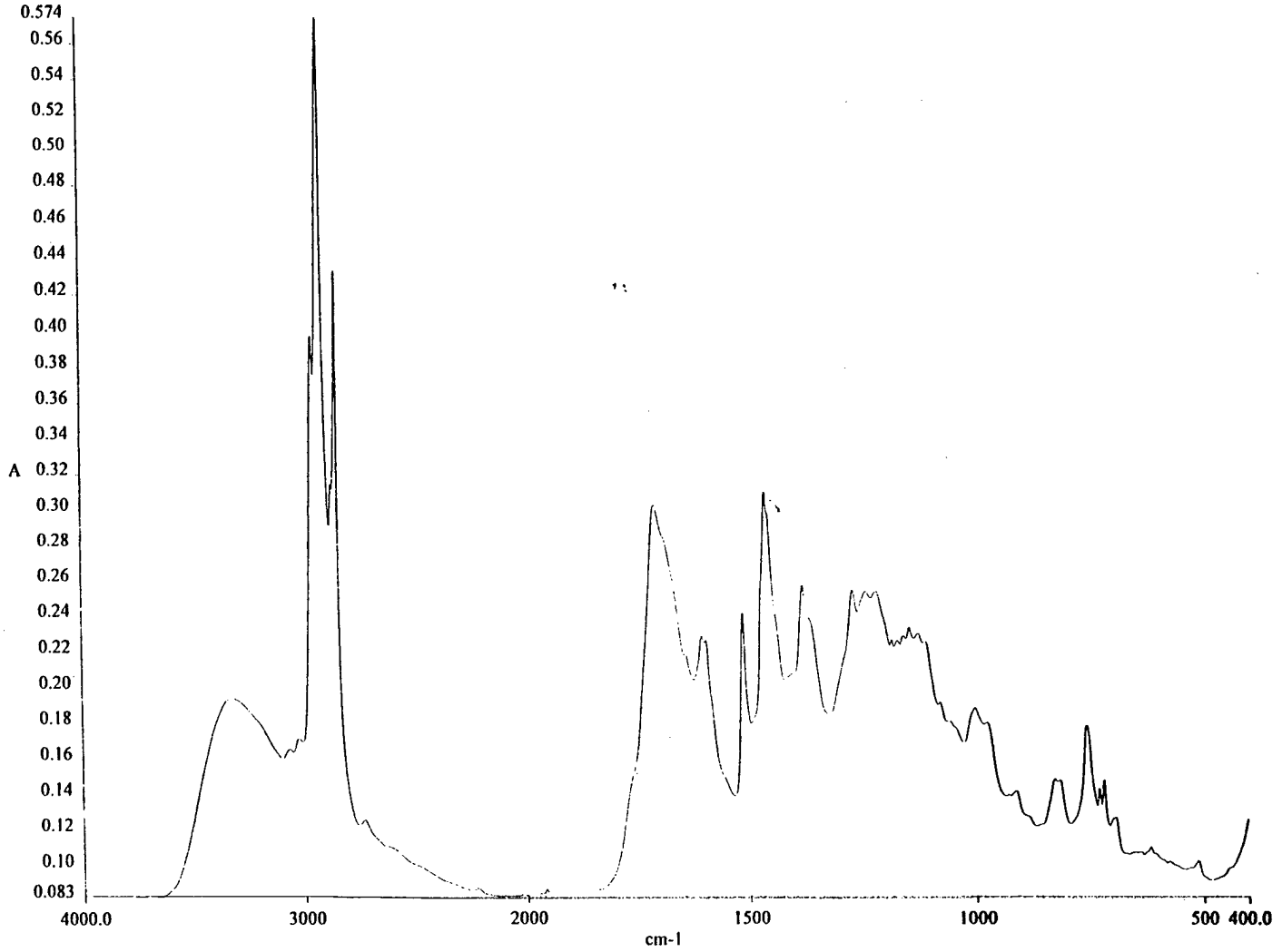

Figure 10. Pyrolosis FT-IR Spectra of Layer 2.

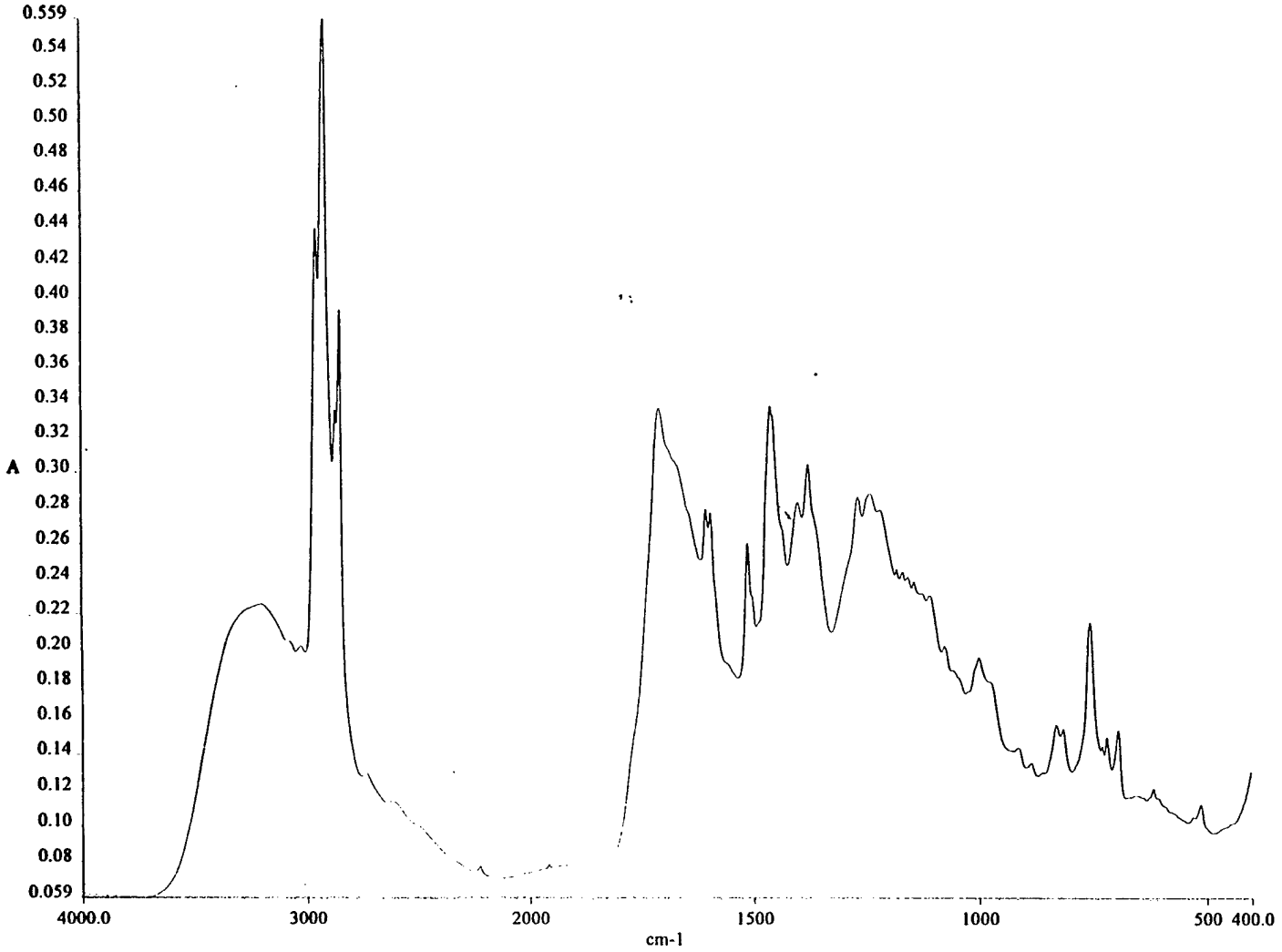

Figure 11. Pyrolosis FT-IR Spectra of Layer 7.

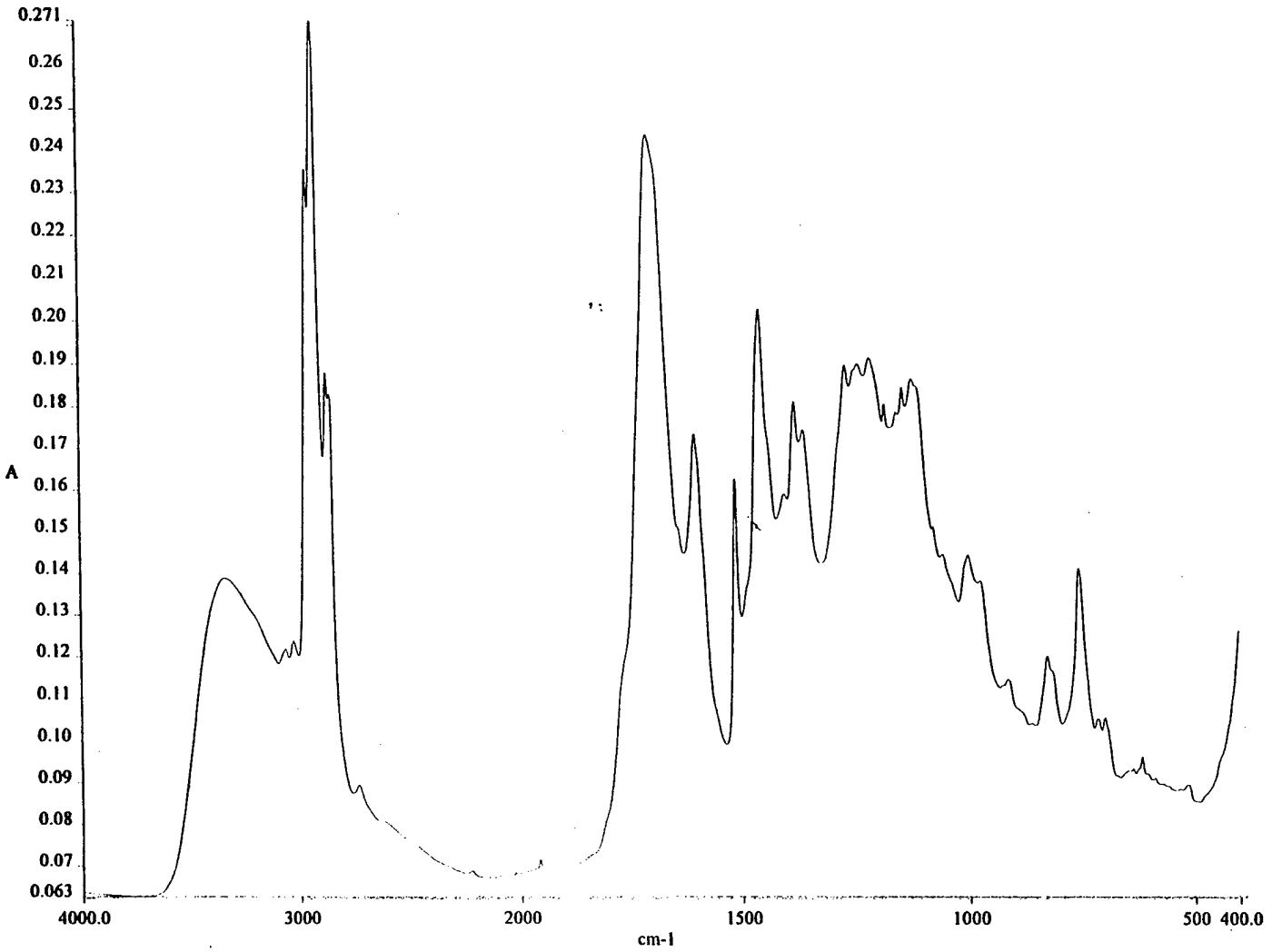

Figure 12. Pyrolosis FT-IR Spectra of Layer 16.

4. Summary

This report has identified and quantified the composition of an unknown cored section of Russian composite armor. The material was identified as a woven glass in an epoxy matrix. The ESEM results determined that the reinforcing fiber is E glass, as opposed to S glass. E glass is inferior to S glass in both armor and structural applications; however, it is often chosen as a reasonable tradeoff because it is much more affordable. FT-IR, results identified the matrix to be an epoxy resin—likely a diglycetal ether of bisphenol-A. TGA clearly showed that all specimens contain approximately 90 weight-percent fiber and 10 weight-percent organic resin. The cored composite density was examined. All density measurements were close to 2.0 g/cm^3, which is consistent with the typical density of a highly filled glass/polymer composite. It was found that the bulk and local section density values were approximately equal within a reasonable margin common in the composite materials industry. No designed-in density gradient vs. length was observed.

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13. ABSTRACT *(Maximum 200 words)*

The chemical and physical characterization of a foreign polymer composite of Russian armor was performed. This report has identified and quantified the composition of an unknown cored section of Russian composite armor.

The material was identified as a woven glass in an epoxy matrix. The environmental scanning electron microscopy (ESEM) results determined that the reinforcing fiber is E glass, as opposed to S glass or the Russian equivalent of S glass, Vertex. E glass is inferior to S glass in both armor and structural applications; however, it is often chosen as a reasonable tradeoff because it is much more affordable. Fourier transform infrared spectroscopy (FT-IR) results identified the matrix to be an epoxy resin—likely a diglycidyl ether of bisphenol-A. Thermal gravimetric analysis (TGA) clearly showed that all specimens contain approximately 90% fiber and 10% organic resin by weight. The cored composite density was also examined. All density measurements were close to 2.0 g/cm^3, which is consistent with the typical density of a highly filled glass/polymer composite. It was found that the bulk and local section density values were approximately equal within a reasonable margin common in the composite materials industry. No designed-in density gradient vs. length was observed.

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